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Degradation characteristics of secondary effluent of domestic wastewater by combined process of ozonation and biofiltration

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Abstract

The performance of the combined process of ozonation and biofiltration was studied for treating the secondary effluent from sewage treatment plant. It was found that COD, NH_3–N, and TOC were removed from 40–52, 10–19, and 9–13 mg/L in the raw water to 18–23, 0.5–1.5, and 7–8.5 mg/L in the effluent water (removal efficiency were 58, 89, and 25%, respectively), respectively, with an ozone dose of 10 mg/L (0.7–1.1 mg O_3/(mg TOC) and 0.2–0.25 mg O_3/(mg COD)), and contacting time of 4 min. Under the operation conditions, ozonation enhanced the biodegradability of the organics in the secondary effluent, as illustrated by increasing biodegradable dissolved organic carbon (BDOC) value from 0.8–1.1 mg/L in the raw water to the 2.0–2.7 mg/L in the effluent water. Meanwhile, the percentage of the organics with molecular size less than 1 k Da in the secondary effluent increased from 52.9 to 72.6%. The experimental results supported the expectation that the combined process of O_3/Biofiltration might enhance the overall treatment efficiency of secondary effluent treatment.

Keywords: Ozonation; Biofilter; Advanced treatment; Secondary effluent of domestic wastewater

1. Introduction

Because of the increasing pressures of wastewater discharge on water supplies and the shortage of water resources, more and more attentions have been paid on the reuse of secondary effluent of municipal wastewater [1,2]. Especially, China is also facing serious water shortage problems [3,4], and advanced treatment of wastewater for reuse is thus in urgent need. The process of coagulation–flocculation–clarification–filtration is one of the well-known conventional processes for advanced treatment of municipal wastewater. However, it seems to have some obvious disadvantages such as large land occupation, complicated operation and sophisticated management despite of its acceptable treatment performance. Whereas, other attractive advanced treatment process such as microfiltration (MF) or ultrafiltration (UF) technology, which are in widespread use in the west countries, may be difficult to use at large in developing countries such as China at present, due to its possible higher costs. Therefore, it seems necessary to develop relatively low cost processes (including some combined processes) with relatively higher treatment efficiency.

Chemical oxidation can degrade organic compounds without any additional waste produced. The most common oxidants are ozone and H_2O_2 etc. Ozone is a very powerful oxidant (Redox potential 2.07 V for ozone *versus* 2.8 V for hydroxyl radical) for water and wastewater treatment. Once dissolved in water, ozone reacts with a large number of organic compounds in two possible ways: direct oxidation, as molecular ozone, or indirect reaction through the formation of secondary oxidants such as free radicals, particularly hydroxyl radical. Ozone treatment of several types of wastewaters, resulting in considerable organics elimination, had been reported by some researchers [5–7]. Furthermore, ozonation is expected to cause an increase of the biodegradable organic carbon for subsequent biological stages [8–10].

Biofilter originated in late 1980s and early 1990s in Europe [11], and is considered as an alternative to the traditional acti-

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vated sludge process, which is commonly used in biological wastewater treatment. The primary advantages of the process are a reduced footprint resulting from the elimination of secondary clarifiers and their associated operational difficulties, and a low hydraulic retention time due to high biomass retention in the system. More important, less surplus sludge was produced during the operational period, which thus enormously reduced the surplus sludge treatment costs. Attentions about biofilters have been paid in wastewater treatment field [12–15] in the last two decades.

In general, biodegradable COD proportion in the secondary effluent of sewage, which can be indicated by biodegradable dissolved organic carbon (BDOC) is relatively low. In order to improve the treatment efficiency, it is necessary to increase BDOC of the influent to be treated biologically. As a powerful oxidant, ozone can transform some refractory organic matters to biodegradable organic ones, that is, BDOC, which can be removed more easily through biodegradation. The combined process of ozonation and biological treatment is one of the most promising processes among advanced treatment processes [16]. Based on the above description, chemical oxidation (ozonation in this study) as pretreatment unit of biochemical treatment (biofilter in this study) is necessary as well as promising for improving the treatment efficiency of secondary effluent.

The aim of the present study is to investigate the characteristics of degradation/conversion of organics, especially biorefractory organics, by combining preozonation with biofiltration for the treatment of secondary effluent from domestic wastewater treatment plant.

2. Material and methods

2.1. Water characteristics

The raw water in this study was the secondary effluent obtained from a conventional activated sludge system in Wenchang Municipal Wastewater Treatment Plant of Harbin in China, in which the anaerobic/aerobic (A/O) process was used. The raw water was firstly filtered by sand column with 300 mm in height to avoid the negative impact of suspended solid (SS) on the laboratory study, because the removal characteristics of the dissolved organics were focused in this study. The secondary effluent quality after filtration is shown in Table 1.

Table 1
Characteristics of the secondary effluent fed to ozonation/biofiltration system

Parameter	Range	Average
COD (mg/L)	40–55	52.5
BOD (mg/L)	11–15	13
TN (mg/L)	20–25	22.5
NH_3–N (mg/L)	10–20	13
TOC (mg/L)	9–13	11
UV_{254} (cm^{-1})	0.16–0.22	0.19

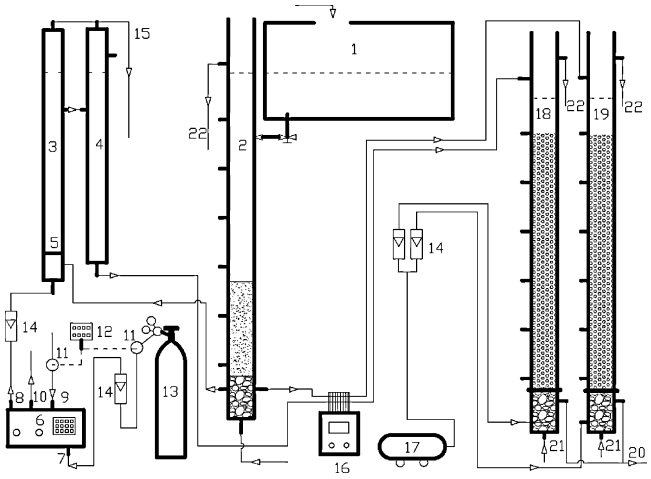

Fig. 1. The schematic diagram of the ozonation/biofiltration process (1) feed tank; (2) sand filter column; (3) ozonation reaction column; (4) retention column; (5) gas diffuser; (6) ozone generator; (7) oxygen feed inlet; (8) ozone outlet; (9) cooling water inlet; (10) cooling water outlet; (11) electromagnetic valve; (12) automatic controller; (13) oxygen bottle; (14) gas flowmeter; (15) outlet of residual ozone gas; (16) peristaltic pump; (17) air compressor; (18) No. 1 biofilter column; (19) No. 2 biofilter column; (20) biofilter outlet; (21) backwashing water inlet; (22) backwashing water outlet.

2.2. The experimental procedure

The schematic of the ozonation/biofiltration system is shown in Fig. 1. It consists of two treatment trains: biofiltration process and ozonation followed by biofiltration process (ozonation/biofiltration). The raw water was filtered by 300-mm high sand (Diameter 1–3 mm) column before it was sent to the ozonation column or pumped directly to the No. 2 biofilter column.

The biofilter columns are 2400 mm in height and 50 mm in inner diameter. The columns contained a clay-based media approximately 2–4 mm in diameter with an average specific surface area of 2–6 m^2/g. A percolated plate was placed in each column to support the 1600 mm media layers. Air was provided into the columns through a diffuser located near the bottom of the columns. The hydraulic flow rates were controlled in the range of 0.433–1.732 $m^3/(m^2/h^{-1})$ with the peristaltic pump. Each column was backwashed at irregular intervals (21–49 days) with combined air (6 L/min) and water (8 L/min) simultaneously.

Ozone was produced from pure oxygen using a DHX-SS-G1 ozone generator (Harbin Jiujiu Electrochemistry Engineering and Technology Company, China). The pure ozone dose was controlled at approximately 10 mg O_3/min for ozonation. During reaction, the ozone dose and residual amount of ozone were monitored to calculate the utilization efficiency and amount of ozone used. The ozonation reactor (glass-made) and retention column have working volume of 1.2 and 1.0 L, respectively. The ozone generator was controlled to work for 1 min every 40 min periodically (during working time, the two electromagnetic valves were of start-up to open the oxygen feed and the cooling water, respectively. At other time, both of the valves are shut down controlled automatically by the controller according to the flow rate of the influent and retention time. The amount of ozone used was 0.77 to 1.1 mg O_3/(mg TOC) and 0.2–0.25 O_3/(mg

COD), in other words, 10 mg O_3/L in wastewater. The total ozonation time is 4 min as mentioned above. The residual ozone dissolved in water was removed in the retention column by aeration.

2.3. Analytical procedures

The concentration of ozone was monitored with the two methods. The absolute ozone amount in the inlet and outlet gas streams was determined with titration method [18] using Kalium Iodide and $Na_2S_2O_3$. The residual ozone in liquid phase was directly determined at 258 nm using a Shimadzu UV-2550 spectrophotometer. The absorbance values for ozone were converted in concentration units using a molar absorption coefficient for ozone 3000 M^{-1}/cm [17]. The comparison of the above two methods indicated that they had the same precision.

The water samples to be analyzed were collected from all the sampling ports regularly every 2–5 days (except for two weeks of 2006 Spring festival days) from September 2005 to April 2006. The following items are monitored regularly: COD, NH_3–N, and color were determined with closed reflux micro methods, Nessler's reagent colorimetric method and Pt–Co standard colorimetry method [18], respectively. TOC and TN were monitored with Shimadzu TOC/TN-5000 analyzer (Japan), UV_{254} was determined with Shimadzu UV-2550 spectrophotometer (Japan). Other items such as temperature, dissolved oxygen (DO) (by DO analyzer, model 55, YSI Incorporated, USA), and pH were routinely monitored during the whole experimental period.

BDOC of the water was determined according to the reference [19]. The sample to be analyzed was filtered through 0.45 μm membrane, and then the TOC was determined. An aliquat of 1 ml of inoculums-water was induced to 100 ml of the filtered water mentioned above. Then, the sample with inoculums was filtered by 0.45 μm memabrane after 5-day culture at 20 °C, and the TOC was determined, too. The difference of TOC value before and after the culture is considered as the BDOC of the water.

Additionally, the determination of molecular size distribution (MSD) of the organics in the influent and the effluent of the system was carried out with stirred ultrafiltration cell (model 8400, Millipore, USA) combined with TOC analyzer. The operational steps are as follows: First, the water sample was filtrated using a membrane of 0.45 μm pore size. Then, the subsequent ultrafiltration operation (Driven by 0.5-MPa high-pure N_2) was carried out using the stirred ultrafiltration cell, with different pore size membranes, such as 1, 3, 5, 10, and 100 k Da, respectively. Third, TOC of the filtrate were analyzed. Fourth, the MSD were calculated based on the TOC values. Specially, TOC mass balance was made and the results indicated that the total TOC is equal to the sum of every partial TOC in different MSD ranges considering the error factor.

The color of the water sample was determined as follows [18]. A series of Pt–Co color standard solutions were prepared, and determined consequently using UV spectrophotometer at 420 nm. Then, a linear regression equation of absorbance against color can be obtained: $Y = 0.0004X + 0.0021$ with an R^2 of 0.9998. (Y is the absorbance and X is the color value. The valid range of color is from 0 to 100° in this experiment).

3. Results and discussion

3.1. Start-up of biofiltration system and biofilm formation

The start-up of biofiltration system (without ozonation) was extended for about five weeks (from September 15th to October 19th, 2005) as shown in Fig. 2(A) and (B).

Due to the low microbial population and nutrient concentration of the secondary effluent, a previous inoculation of biological filter is necessary in order to obtain an adequate biofilm yield. In the operational period, the sludge from the second settlement tank of Wenchang wastewater treatment plant was used as seeding sludge. In the first three days, the static culture was carried out using the fresh domestic wastewater as feed (COD, 350–400 mg/L, NH_3–N, 30–40 mg/L). After three days static culture, the continuous stream culture was undertaken subsequently with the mixture of the domestic wastewater mentioned above and the secondary effluent. It is worthy to note that the ratio of the later to the former is increased gradually until the later became the only feed in the sequent cultural days. As shown in Fig. 2(A), the COD removal efficiency lowered continuously and stabilized for 25% after two cultural weeks, and it was resulted from the much low organic loading of the secondary effluent. During the cultural days, the NH_3–N removal efficiency maintained fluctuant going-up in 25 days, when the removal efficiency was stabilized for 85% or so. The biofilm formation after inoculation was confirmed by the scanning electron microscopy (SEM). When activated sludge was used as inoculum, a heterogeneous biofilm was obtained, showing sev-

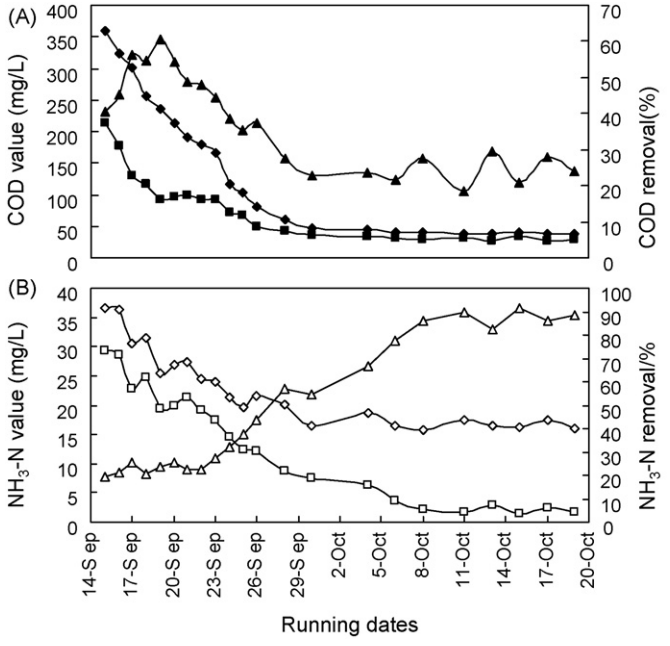

Fig. 2. Start-up profile of the biofiltration system (A) (♦) influent COD, (■) effluent COD, (▲) COD removal efficiency; (B) (◊) influent NH_3–N, (□) effluent NH_3–N, (△) NH_3–N removal efficiency.

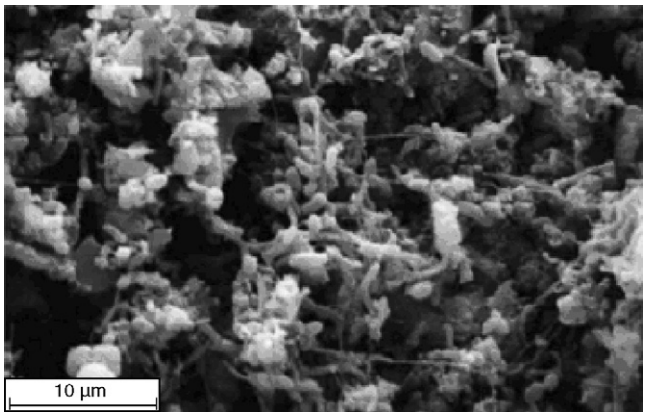

Fig. 3. Scanning electron micrographs (1000×) of biofilm formed after inoculation using activated sludge (biomass amount was 59 nmol P/(g media)).

eral bacterial morphologies and microbial types (Fig. 3). The properties of biofilms did not significantly vary during the operation time of the bioreactor (14 days after reaching steady state conditions) by possible reasons of inoculants, and the feed water being from the same original system.

Biomass amount was 50–60 nmol P/(g media) in the beginning media layer (15-cm-high) of the biofilter column at pH 6.7–7.8. However, with the ozonation, there seemed much more organic base bioavailable for microorganisms due to the improvement of BDOC of the influent. Thus, at the parallel processing conditions, it is found the biomass amount was increased to 58–68 nmol P/(g media) (15-cm-high beginning media layer) of the biofilter column after ozonation. More experiments are in progress (using DGGE technology) in order to understand in detail the characteristics of bacteria on the films formed by inoculation, and the influent of alteration in microbial composition over quality of treated effluent.

3.2. The optimal ozonation time

At an ozone dose of 10 mg/L (0.25 mg O_3/(mg COD), and (0.94 mg O_3/(mg TOC)), the COD and TOC reduction of the secondary effluent by ozonation are demonstrated in Fig. 4. It is obvious that COD and TOC are reduced from 38.2 and 10.92 mg/L, respectively in the raw water to 29.6 (25.7% eliminated) and 8.11 mg/L (16.5% eliminated), in the effluent,

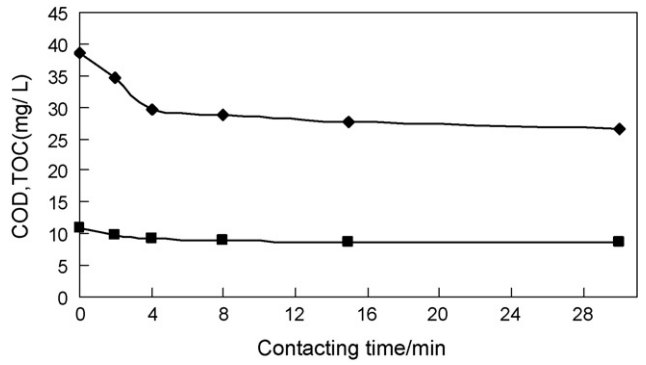

Fig. 4. The removal of COD and TOC by ozonation (♦) COD; (■) TOC. Ozone dose, 10 mg/L (0.916 mg O_3/(mg TOC), 0.25 mg O_3/(mg COD)).

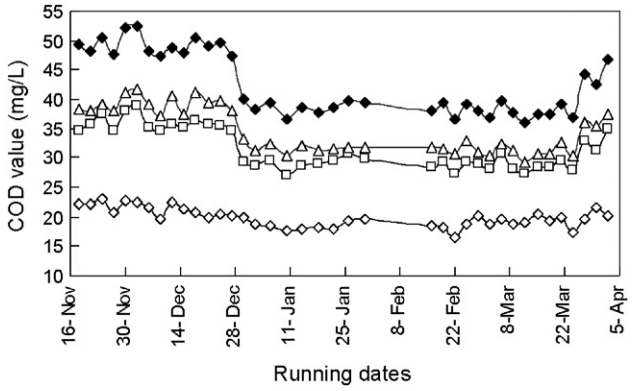

Fig. 5. COD removal by ozonation, biofiltration, and combined ozonation and biofiltration, respectively (■) COD of the influent; (□) COD in the effluent in ozonation alone; (△) COD after biofiltration alone; (◊) COD in the effluent of combined ozonation and biofiltration process. Ozone dose: 10 mg/L (0.2–0.25 mg O_3/(mg COD)), contacting time: 4 min.

respectively, at 4 min of contacting time. However, only 5% increase of removal efficiency can be found when the contacting time was extended to 30 min. It indicates that 4 min can be considered as the optimum contacting time at an ozone dose of 10 mg/L for the treatment of this water and this contacting time ensures the highest efficiency of ozone utilization.

3.3. Degradation characteristics of secondary effluent by ozonation/biofiltration

The degradation characteristics of the secondary effluent of the domestic wastewater by ozonation and ozonation/biofiltration processes were studied for six months or so. The results are shown in Figs. 5 and 6, respectively. From Fig. 5, it is noted that the removal efficiency of COD by ozonation alone and by biofiltration alone maintains 20–30 and 17–25%, respectively. However, the total removal efficiency was enhanced to 46–63%, when biofiltration was combined with preozonation, which is higher than the sum of the former two. The combined removal is partly attributed to the direct oxidation by ozone. More important is that ozonation increased the biodegradability

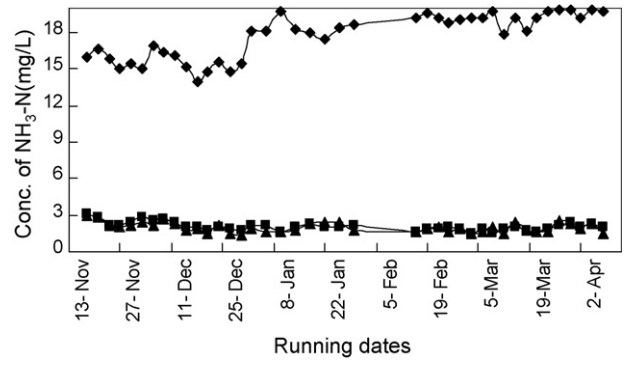

Fig. 6. NH_3–N removal by biofiltration alone and the combined process of ozonation and biofiltration, respectively. (♦) influent NH_3–N; (▲) effluent of biofiltration alone; (■) effluent of combined process of ozonation and biofiltration. Ozone dose: 10 mg/L (0.77–1.1 mg O_3/(mg TOC), 0.2–0.25 mg O_3/(mg COD)), contacting time: 4 min.

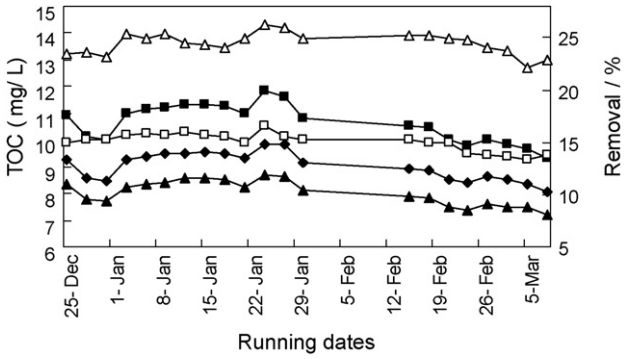

Fig. 7. TOC removal by ozonation and ozonation/biofiltration process, respectively (■) TOC of the raw water; (♦) TOC of ozonized water; (▲) TOC of ozone/biofiltration effluent. TOC removal of ozonized water; (△) TOC removal in the ozone/biofiltration process. Ozone dose: 10 mg/L (0.77–1.1 mg O_3/mg TOC), contacting time: 4 min.

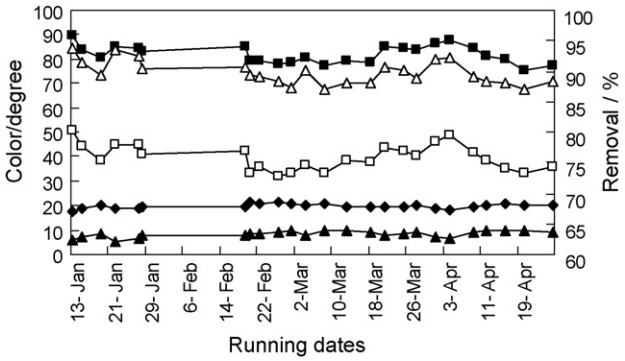

Fig. 9. Color removal by ozonation and ozonation/biofiltration process, respectively (■) color of feed water; (♦) color of ozonized water; (▲) color in the effluent of the ozonation/biofiltration process; (□) color removal of ozonized water; (△) color removal of in ozonation/biofiltration process. Ozone dose: 10 mg/L (0.77–1.1 mg O_3/(mg TOC)), contacting time: 4 min.

of the influent, which increased effectively the removal efficiency of the sequent biofiltration unit.

From Fig. 6, it is noted that the average removal of NH_3–N reached 90% by the biofiltration alone as well as ozonation/biofiltration at the experimental ozone dose and contacting time. It is obvious that the removal of NH_3–N is dominant via biological function in the biofiltration system and the removal cannot be enhanced by preozonation. Moreover, during the same experimental period, it is found that 80–90% of NO_2^-–N was removed by ozonation alone before biofiltration unit. Nitrification also made the concentration of NO_3^-–N vary from 3–5 mg/L in the influent to 15–20 mg/L in the effluent in biofiltration alone as well as the ozonation/biofiltration process.

The TOC removal results are shown in Fig. 7. The original TOC of the domestic secondary effluent ranged from nine to 13 mg/L. It can be seen that the removal efficiency reached 15% or so by ozonation alone. However, it is increased to 25% by the combined process of ozonation and biofiltration. Ozonation can increase the biodegradability of the water, which enhanced the efficiency of the biological treatment (see the later part), even though the organic matters cannot be mineralized thoroughly by ozonation alone.

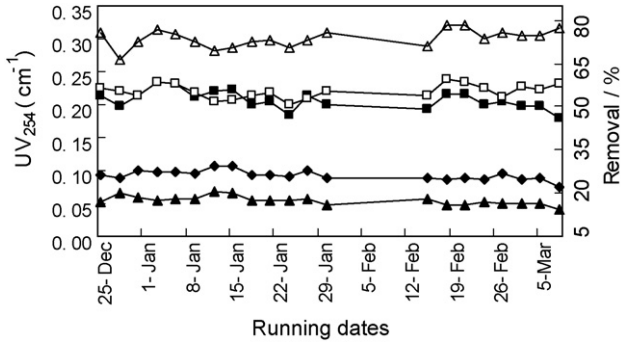

Fig. 8. UV_{254} removal by ozonation and ozonation/biofiltration process, respectively (■) UV_{254} of the raw water; (♦) UV_{254} of ozonized water; (▲) UV_{254} of ozonation/biofiltration effluent; (□) UV_{254} removal of ozonized water; (△) UV_{254} removal in ozonation/biofiltration process. Ozone dose: 10 mg/L (0.77–1.1 mg O_3/(mg TOC)), contacting time: 4 min.

The removal of UV_{254} and color of the feed water are shown in Figs. 8 and 9, respectively. As known, UV_{254} represents the organic matters with C=C, C=O structures, which have strong absorbance at 254 nm. Examples are phenolic, poly aromatic hydrocarbons (PAHs), aromatic ketone, and aromatic aldehyde etc. In general, these compounds have phenyl structures. From Figs. 8 and 9, it can be seen that the removal efficiency of the UV_{254} and color are 60 and 80%, respectively, by ozonation alone. Furthermore, both of them are increased to 75 and to 90% by the combined process of ozonation and biofiltration. The results indicated that most of the C=C, C=O structural group of the organics can be destroyed by ozonation alone. It therefore proved that ozonation contributed much on the removal of UV_{254} and color of the water.

3.4. Effect of ozonation on biodegradability of the secondary effluent

BDOC represents the organics, which can be mineralized by heterogeneous bacteria in water. In fact, BDOC is considered as essential nutrient and energy for metabolic activity of bacteria. In general, the less the BDOC value is, the more disad-

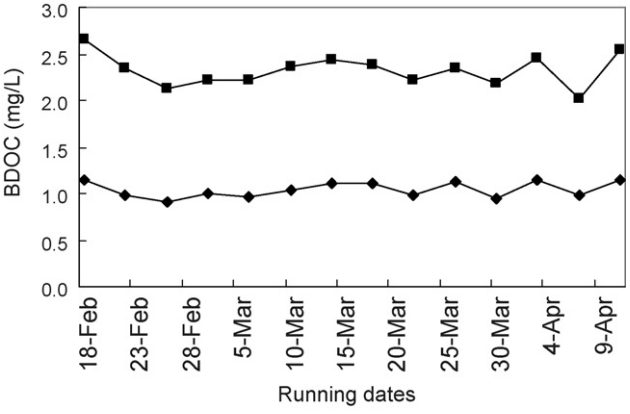

Fig. 10. . Effect of ozonation on BDOC value. (♦) BDOC of feed water; (■) BDOC of ozonized water. Ozone dose: 10 mg/L (0.77–1.1 mg O_3/(mg TOC)), contacting time: 4 min.

Table 2
Effect of ozonation on the MSD of organics in the secondary effluent

Range of molecular weight (Da)	Fed water		Ozonized water	
	TOC (mg/L)	Rate (%)	TOC (mg/L)	Rate (%)
<1 k	6.298	52.86	9.31	72.73
1–3 k	0.726	4.09	1.28	9.95
3–5 k	0.759	3.57	0.10	3.78
5–10 k	2.758	21.45	0.86	6.69
10–100 k	1.269	9.87	0.76	2.91
>100 k	1.050	8.17	0.25	1.94
Sum	12.86	–	12.56	–
<045 μm	12.69	100	12.64	100

Note: ozone dose is 10 mg/L (0.9 mg O_3/mg TOC), contact time is 4 min.

vantaged bacteria will reproduce. Vice versa, it indicates that the organic matter in water is more easily biodegraded. The effect of ozonation on BDOC is shown in Fig. 10. It can be seen that the BDOC values were increased from original 0.8–1.1 mg/L to 2.0–2.7 mg/L by ozonation within 4 min contacting time at an ozone dose of 10 mg/L, that is, 50% improvement was realized. This result also indicated that ozonation can significantly improve the biodegradability of the secondary effluent, in other words, the biochemical treatment efficiency can be much improved by taking ozonation as pretreatment unit.

3.5. Effect of ozonation on MSD of the secondary effluent

It is believed that MSD of organic matter can reflect the biological degradation characteristics of the wastewater to be treated. In general, the higher-MS-organics should be transformed to the lower-MS-organics before microorganisms can degrade it efficiently.

The effect of ozonation on MSD of the organics in the secondary effluent is shown in Table 2. We can see that the molecular size of most of the dissolved organics is less than 1 k Da (52.86%), or range from 5 to 10 k Da (21.4%). However, the ozonation within 4-min contacting time makes the MS distribution change significantly at ozone dose of 10 mg/L. That is, the organics rate of MS < 1 k Da was increased to 72.73% from original 52.9% and the rate of 1 < MS < 3 k Da was improved to 9.95% from original 4.09%. To some extent, the changing of MSD induced by ozonation explains the improvement of removal efficiency of biofiltration followed by ozonation.

4. Conclusions

In this study, the degradation characteristics of the secondary effluent of domestic wastewater by the combined process of ozonation and biofiltration was undertaken. It shows that the removal efficiencies of COD, NH_3–N, TOC, UV_{254}, and color of the secondary effluent reached 58, 90, 25, 75, and 90%, respectively, at an ozone dose of 10 mg/L (0.7–1.1 mg O_3/mg TOC) and ozonation time of 4 min. Ozonation can enhance the biodegradability of the secondary effluent by improving BDOC value from original 0.8–1.1 mg/L to final 2.0–2.7 mg/L. Meanwhile, the percentage of the organics with molecular size less than 1 k Da in the secondary effluent increased from original 52.9% to final 72.6%, for 4-min ozonation time, and it therefore supported the explanation of the upgraded performance of the combined process of ozonation/biofiltration to a certain extent.

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